



## Record of Invention

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Information

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### **Cosmetic Compositions for the Skin and the Hair Based on Acrylic Polymers That Contain Hydrophobic Cyclic Monomers**

#### **Investigator Information**

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#### **Description of the Invention**

According to the present invention there is provided cosmetic compositions for skin and hair in the form of an emulsion or dispersion comprising (a) a copolymer of (i) a (meth)acrylate ester of C4 to C18 straight or branched chain alkyl alcohol, (ii) a (meth)acrylate ester of a saturated or unsaturated cyclic alcohol containing 6 to 20 carbon atoms, and (iii) optionally up to 10% by weight of a hydrophilic (meth)acrylate monomer; and (b) an aqueous carrier, solvent, or vehicle component. The cycloalkyl (meth)acrylate monomer (component (a)(ii)) is preferably one whose homopolymer has a glass transition temperature (T<sub>g</sub>) of about 30° C or greater and is selected from the group consisting of phenyl acrylate or methacrylate and analogues substituted with alkyl groups including tolyl and t-butylphenyl (meth)acrylate, bicyclo[2.2.1]heptyl acrylate or methacrylate and analogues substituted with alkyl groups including isobornyl (meth)acrylate, adamantyl acrylate or methacrylate and analogues substituted with alkyl groups including 3,5-dimethyladamantyl (meth)acrylate, benzyl methacrylate, cyclohexyl methacrylate and analogues substituted with alkyl groups including menthyl methacrylate and 3,3,5-trimethylcyclohexyl methacrylate, dicyclopentenyl acrylate or methacrylate, 2-(dicyclopentenyl)oxyethyl acrylate or methacrylate, and mixtures thereof. The amount and nature of components (a)(i), (ii), and optionally (iii) is chosen to give an aqueous copolymer emulsion or dispersion which on drying forms a flexible non-sticky film possessing good cohesive strength.

#### **Utility of Invention**

Cosmetic compositions with improved abrasion resistance; transfer resistance; water, perspiration, and humidity resistance; and excellent gloss, feel, and adhesion are provided which dry quickly and are non-sticky.

## Background

The use of polymers to prevent transfer of makeup or wash off of sunscreen is known. Generally these polymers are hydrophobic in nature and obtain their hydrophobicity from long chain alkenes (for example, ISP's GANEX resins described in US 5,026,540; US 5,171,807; US 5,219,559; US 5,516,508; US 5,518,712; and US 5,547,659) or long chain alkyl (meth)acrylates (for example, those described in US 4,172,122 and US 4,552,755). Also claimed as useful for this purpose are silicone pressure sensitive adhesives (US 5,460,804), styrene-ethylene-propylene block copolymers (US 6,060,072), or polymers containing long chain vinyl or allyl ester comonomers (US Re. 29,871). Such polymers have a low glass transition temperature and can leave skin with a sticky or tacky quality. They may also possess poor cohesive strength giving a greasy feel and causing transfer or staining. Cosmetics or sunscreens formulated with them may be harder to smoothly and uniformly apply due to draginess and can give a leaden skin feel, particularly in lipstick.

High glass transition temperature polymers for use in cosmetics are known, particularly as hair styling aids and nail lacquers. For hair styling generally these polymers are glassy because of high levels of polar monomers, such as monomers containing acid, amide, amine, or hydroxyl functionality (see for example US 5,019,377). The use of high levels of polar monomers in the polymer detracts from the polymer's ability to provide water resistance to cosmetic and sunscreen formulations. Up to 20% of hydrophobic high glass transition monomers, such as isobornyl acrylate, have been used to prepare a terpolymer with polar vinyl ester and alkyl maleate half ester comonomers (disclosed in EP 299,025 and WO98/51266) to provide stability during the suspension polymerization, ensure solubility in commercial alcohol carriers, and minimize tack. Nail lacquers are generally applied from organic solvent and hence hydrophobic polymers are used with nitrocellulose being the most widely used material. US-A 4,762,703 (Abrutyn) discloses an anhydrous nail lacquer composition containing 10 to 40 % by weight of a copolymer which is the reaction product of 5 to 30% by weight of diacetone acrylamide with 60 to 95% by weight of (meth)acrylate esters of a) 5 to 48% straight chain alcohols, b) 5 to 60% cyclic alcohols, c) 1 to 25% higher alkyl alcohols, and d) 1 to 30% alkoxy or aryloxy alkyl alcohols. The use of an aqueous carrier, solvent or vehicle component is not disclosed, nor is the use of these materials in cosmetic and sunscreen compositions for skin and hair.

A need exists for non-sticky hydrophobic polymers in an aqueous carrier, solvent or vehicle which can be readily formulated into cosmetic and sunscreen compositions for skin and hair to provide improved abrasion resistance; transfer resistance; water, perspiration, and humidity resistance; and excellent gloss, feel, and adhesion.

## Examples

### Materials

#### General procedure for producing emulsion polymers

Into a 1 liter Mortonized split resin flask was charged 100 grams of monomers (detailed in

Table I below), 80 milligrams of carbon tetrabromide, 124.7 grams of deionized water, 200 milligrams of potassium persulfate, 64 milligrams of sodium metabisulfite, 1 gram of sodium dodecyl benzene sulfonate, and 2.5 g of Mazon SAM 211 alkylene polyalkoxy ammonium sulfate copolymerizable surfactant. The head was placed on the flask and a thermocouple, nitrogen inlet, and mechanical stirrer attached. The headspace was swept with nitrogen at 1 liter per minute while heating the contents with infra red lamps to 30° C and stirring at 250 rpm. 1 gram of a solution of 28 milligrams ferrous sulfate heptahydrate in 50 grams deionized water was charged, the flask sealed, and a vacuum pulled on the flask three times, breaking it each time with nitrogen. After 15 or 20 minutes an exotherm is noted which peaks 20 to 25 minutes later at 55 to 65° C. Reactor temperature is increased to 75° C and held there one hour, then the resulting latex was filtered through doubled over cheesecloth into a jar. In all cases moderate levels of coagulum were noted around the thermocouple and stirring paddle.

Table I: Monomer Charges Used for Emulsion Polymerization

<b>Example</b>	<b>g 2-EHA</b>	<b>g IBOA</b>	<b>g AA</b>	<b>g MAA</b>
Comparative 1	70	25	0	5
Comparative 2	60	35	5	0
Comparative 3	55	40	5	0
1	60	35	0	5
2	55	40	0	5
3	50	45	5	0
4	50	45	0	5
5	50	40	0	10
6	45	50	5	0
7	45	50	0	5
8	35	60	0	5
Comparative 4	25	70	0	5

2-EHA = 2-ethylhexyl acrylate, IBOA = isobornyl acrylate, AA = acrylic acid, MAA = methacrylic acid

Preparation of Comparative Example 5 - An acrylate terpolymer such as those disclosed in US 4,172,122 and 4,552,755

Into a one liter bottle was charged 280 grams of ethyl acetate, 94.6 grams of isooctyl acrylate, 110 grams of stearyl methacrylate, 15.4 g of acrylic acid, and 0.77 grams of 2,2'-azobis(2-methylbutyronitrile) sold by duPont under the tradename Vazo 67. The resulting solution was purged for 5 minutes with nitrogen at 5 liters per minute, sealed, and tumbled in a water bath at 60°

C for 63 hours. A hazy, moderately thick solution resulted.

Preparation of Comparative Example 6 - Ganex V216 solution.

A 40% solids solution of Ganex V216 (a PVP/Hexadecene Copolymer from ISP) was prepared by dissolving 4 grams of Ganex V216 in 6 grams of ethyl acetate with gentle heating.

Test Methods:

Test methods used to evaluate flexibility (or brittleness) and stickiness (or tack) of coatings prepared from the materials described above are industry standard tests as detailed below.

Preparation of Coatings:

Coatings of the examples were prepared on 1.5 mil (38 micron) polyester film using a knife coater yielding 1.5 mil (38 micron) thick coatings after drying for 10 minutes at 70° C in a forced air oven. These coatings were conditioned for 24 hours at 22° C and 50% relative humidity prior to testing.

Flexibility:

The flexibility of each coating was assessed using ASTM D 4338-97, "Standard Test Method for Flexibility Determination of Supported Adhesive Films by Mandrel Bend". The coated polyester was folded with adhesive side out over a 1/8" (3.2 mm) rod and the development of cracks, fracture, or crazing noted as a failure. All coatings passed this test except for Comparative Example 4 which cracked and flaked off the polyester.

Tack:

The tack of each coating was assessed using ASTM D 2979-95, "Standard Test Method for Pressure-Sensitive Tack of Adhesives Using an Inverted Probe Machine". A Polyken Probe Tack Series 400 Tester was used with a dwell time of 1 second, a contact and removal speed of 1 centimeter per second, and a annular ring weighing 19.8 grams. The 5 millimeter stainless steel probe was cleaned with isopropanol between samples and five replicates were run on each coating and averaged to give the results reported in Table II. Materials useful in formulating cosmetic compositions have a tack value of less than 50 grams, preferably less than 30 grams, most preferably equal to 0 grams. Useful materials will also possess high enough cohesive strength so that they don't cohesively fail, transferring residue to the probe. Comparative Example 6 has too low a cohesive strength.

Table II: Probe Tack and Mandrel Testing Results

<b>Example</b>	<b>Tack (g)</b>	<b>Tg (°C)</b>	<b>Mandrel</b>
Comparative 1	59	-39	Pass
Comparative 2	383	-26	Pass
Comparative 3	274	-19	Pass
1	0	-26	Pass

2	0	-19	Pass
3	29	-9	Pass
4	0	-13	Pass
5	0	-13	Pass
6	0	-4	Pass
7	0	-3	Pass
8	0	15	Pass
Comparative 4	0	39	Fail
Comparative 5	316	-	Pass
Comparative 6	Cohesive	-	Pass

#### Cosmetic Examples

An oil in water body lotion was prepared from the emulsion polymer of Example 1: In separate vessels the components of Phase A and Phase B in Table III were heated to 70° C with mixing. Phase B was added to phase A and homogenized using a high shear mixer. After cooling, a substantive, non-greasy, non-tacky body lotion results. Body lotions from the emulsion polymers of Examples 2, 6, 7, and 8 were prepared in similar fashion.

Table III: Oil in Water Body Lotion

##### Phase A

Mineral Oil	10%
Isopropyl myristate	2%
Glyceryl stearate	3%
Stearic acid	4%
Ceteth 20	1%
Lanolin oil	0.6%

##### Phase B

Deionized water	73%
Ex. 1 Emulsion Polymer	5%
HEC	0.2%
Triethanol amine	1.2%

An oil in water mascara was prepared from the emulsion polymer of Example 4: In separate vessels the components of Phase A and Phase B in Table IV were heated to 70° C with mixing. Phase B was added to phase A and homogenized using a high shear mixer. After cooling, a flake-, smudge-, and water-resistant mascara results. Mascara from the emulsion polymers of Example 8 and Comparative Examples 1 and 2 were prepared in similar fashion as was a control with water in place of the emulsion polymer.

Table IV: Oil in Water Mascara

Phase A	
Carnuba Wax	10%
Isopropyl myristate	6%
Glyceryl stearate	3%
Stearic acid	5%
Black iron oxide	10%
Phase B	
Deionized water	43.5%
Ex. 4 Emulsion Polymer	20%
PVP	1%
HEC	0.2%
Triethanol amine	1.3%

A portion of each mascara was coated with a knife coater onto 1.5 mil (38 micron) polyester to a dry coating thickness of 2 mil (51 microns). After drying at room temperature for 24 hours, the coatings were qualitatively assessed for smudge resistance, tack, flake resistance, and water resistance. Smudge resistance was judged by rubbing with a finger and seeing how much had transferred to the finger. Tack was judged by pressing a finger down briefly and removing it, seeing how strong a bond was formed to the coating. Flake resistance was assessed by bending and creasing the polyester, seeing if the coating cracked off. Water resistance was judged by suspending a 1" strip of the coated film in an agitated 32° C waterbath for 20 minutes, then assessing the smudge resistance of the still wet coating. Examples 4 and 8 and Comparative 2 formed balls of coating on rubbing in this test, suggesting that the coating integrity was still good, but the adhesion of the coating to the polyester had been compromised by the water. Results are shown in Table V below.

Table V: Qualitative Testing of Mascara Performance

Polymer	Transfer	Tack	Flake	Wet Transfer
None	A lot	Low	Some	Complete
Example 4	None	Low	None	Balls up

Example 8	None	Low	None	Balls up
Comparative 1	Some	Moderate	None	Some
Comparative 2	Some	Moderate	None	Balls up

### Signature Information

*Described by*

Steve S. Kantner

*Signed on:*

August 2, 2000

*This document has been read and understood  
by me:*

Kristin J. Godbey

*Witnessed on:*

August 2, 2000